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Explosives Research and Development Establishment - Rocket Propulsion Establishment



***Explosives Research
and Development
Establishment***

**Technical Note
No. 140**

COUNTED IN

**The Surface Deposition of Graphite on to
RD1355 (Monobasic Lead Azotetrazole)**

J. M. Jenkins
J. R. White

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Technical Note No 140

January 1975

The Surface Deposition of Graphite
on to RD1355 (Monobasic Lead Azotetrazole)

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J M Jenkins

J R White

SUMMARY

A process has been devised to deposit graphite on to the surface of RD1355 (monobasic lead azotetrazole) using a combination of wetting and non-ionic flocculating agents. The product is homogenous, but it is not suitable for use as a conducting composition. The resistance of the pressed compositions are very sensitive to variations in graphite content compared with the equivalent compositions made by the dry mix process.

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1 INTRODUCTION

RD1355 (monobasic lead azotetrazole) has been shown to form a technically useful conducting composition when mixed with small percentages of graphite. This composition is now in British Service in certain conducting composition detonators.¹ Despite the successful use of the mix, the Ordnance factories have reported difficulty in obtaining homogenous mixes and reproducible performance between mixes. Considerable effort has to be devoted to ensuring that each batch is suitable for filling into the store. It was suggested by ERDE that the problem might be solved by a process in which the graphite is deposited on to the surface of the RD1355 crystals. A procedure for this had been devised for RD1303 (lead styphnate) using gelatin and surface active agents.^{2,3} However, this process also coated the RD1303 crystals with gelatin as well as the graphite which introduced problems of hygroscopicity and variability. The gelatin coating can be clearly seen by comparison of Figs 1 and 2. It was hoped that a non-gelatin process could be devised for the coating of the RD1355.

2 OBJECT

To develop a process to give a reproducible RD1355/graphite mix suitable for use as a conducting composition.

3 EXPERIMENTAL

Monobasic lead azotetrazole (RD1355) was manufactured by the specified process.⁴ The graphite (DOHM'S air floated) surface active agents and flocculating agents were purchased from trade. The deposition process was developed on the laboratory scale then scaled up to the semi-technical scale. An example of each scale of preparation is given below.

Laboratory scale

6 g RD1355 was stirred in 150 ml of aqueous 0.2% solution of Johnsons 326 wetting agent for 5 minutes, together with the appropriate weight of graphite to give the desired percentage. A 0.1% solution of Glovers Polyteric EN4 was added dropwise until complete deposition was attained. (2 ml usually required.) The clear mother liquor was decanted off and the product washed once by decantation. The product was collected on a filter and further washed once with water and once with ethanol. It was dried at 40°C and passed through a 60 mesh sieve.

Semi-technical Scale

As above but with the following quantities:

110 g of RD1355, 1500 ml of wetting agent solution, 21 ml of flocculating solution.

Compositions were made with the following graphite percentages:

1, 2, 2.3, 2.5, 2.7 and 3.0.

It was shown that Tween 20 would also act as a satisfactory wetting agent. Cationic and anionic flocculating agents were tried but only the non-ionic EN4 was successful.

4 RESULTS AND DISCUSSION

4.1 Analysis

Gravimetric analysis of the RD1355/graphite mixtures showed that the graphite was evenly distributed throughout the mass of the composition. A typical set of results for a semi-technical batch (WBW 85) is as follows: Graphite contents of 3.16%, 3.21%, 3.14%, 3.15% were obtained.

4.2 Scanning Electron Microscopic Examination

The surface deposited composition was compared with a sample of RD1355 and a dry mixed composition in the scanning electron microscope. The graphited compositions both contained 3% (nominal) graphite. The results are shown in Figures 3 to 8. Figures 3 to 5 show the compositions at relatively low magnification (X144). It can readily be seen that the wet mixed surface deposited sample (Fig 5) is more completely coated than the dry mixed composition (Fig 6). At higher magnification (X720) it can be seen that there are large aggregates in the dry mix and that the particle size of the graphite is generally smaller in the wet mixed composition. (Figs 7 and 8). It can also be seen that there is little or no flocculating agent attached to the crystals of RD1355.

4.3 Sensitiveness Tests

4.3.1 Mechanical Sensitiveness

There is no significant difference between the pure RD1355, the dry mix and the surface deposited wet mix. All three are sensitive initiating compositions.

4.3.2 Electrical Sensitiveness

There is no advantage with the surface deposited compositions compared with the corresponding dry mix. The minimum ignition energy for the surface deposited graphite increases from 250 ergs (1% graphite) to 450 ergs (2%) to 2500 ergs (3% graphite). The value for RD1355 is 125 ergs and for the corresponding 3% mechanical mix is 2400 ergs.

4.4 Electrical Resistance Tests

The electrical resistance tests were carried out in the standard N8 igniter bodies by RARDE (EM1). The required resistance range is 60 to 100 ohms.

Nominal graphite (%)	Resistance range (ohms)
	Compositions pressed at 275 lb dead load
1.0	∞
2.0	1950 - ∞
2.3	640 - 45,000
2.5	195 - 1150
2.7	98 - 1800
3.0	5 - 6

These results show that there is a very sharp drop in resistance between 2.7 and 3.0% graphite. There is not such a dramatic effect with the dry mixes. In practical terms it would be unrealistic to try to devise a process which could give a reproducible composition when the graphite % v resistance curve is so steep. Minor variations in the graphite percentage as would be experienced in production would give rise to large variations in resistance.

5 CONCLUSIONS

5.1 It has been shown that graphite can be successfully deposited on to RD1355 crystals by a wet process using a combination of wetting and non-ionic flocculating agents.

5.2 The mechanical sensitiveness of the surface deposited material is the same as the conventional graphite/RD1355 dry mixes and RD1355. There is an improvement in the sensitiveness to electrostatic discharge compared with RD1355 alone but no improvement compared with the equivalent dry mix.

5.3 The product of the wet process is not suitable for current UK designs of conducting composition devices.

6 RECOMMENDATIONS

No further work be done on surface deposited RD1355/graphite compositions.

7 ACKNOWLEDGEMENTS

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J Roberts and W B Scamaton for their assistance with this work.

8 REFERENCES

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FIGS. 1,2

SCANNING ELECTRON PHOTOMICROGRAPHS

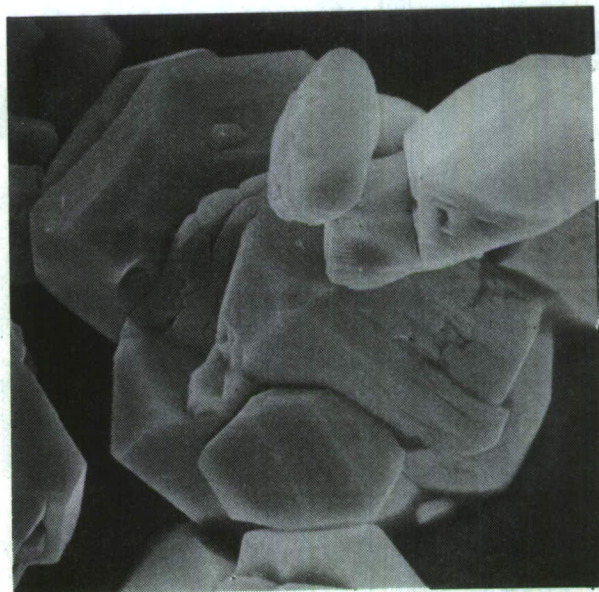


FIG. 1 LEAD STYPHNATE (RD1303) (X720)

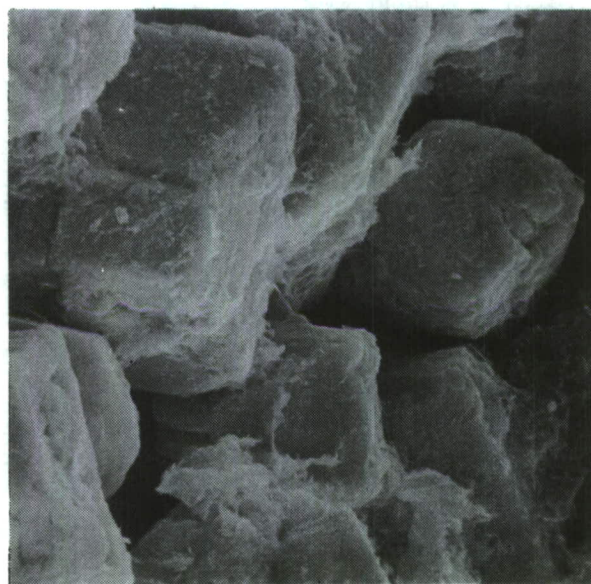


FIG. 2 LEAD STYPHNATE (RD1303)
GRAPHITE/GELATIN COATED (X720)

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FIGS. 3,4,5



FIG.3 MONOBASIC LEAD
AZOTETRAZOLE (RD1355) (X144)



FIG.4 MONOBASIC LEAD
AZOTETRAZOLE (RD1355) WITH
3% GRAPHITE DRY MIXED (X144)

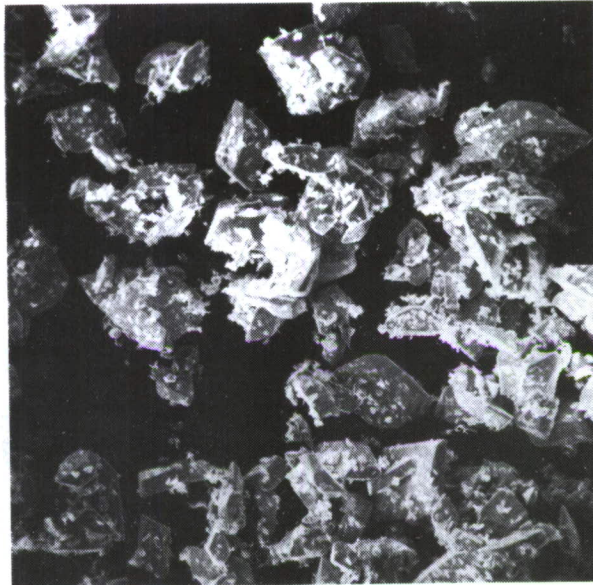


FIG.5 MONOBASIC LEAD AZOTETRAZOLE (RD1355)
WITH 3% GRAPHITE SURFACE DEPOSITED
(X144)



FIG. 6 RD1355 (X720)



FIG. 7 RD1355 3% GRAPHITE
DRY MIX. (X720)



FIG. 8 RD1355 3% GRAPHITE SURFACE
DEPOSITED (X720)

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